PREPARATION OF CERAMIC POROUS BODY USING DIFFERENT VOLUME CONCENTRATION OF BALL CLAY & WHEAT FLOUR

A thesis submitted in the partial fulfillment of the requirements for the degree of

BACHELOR OF TECHNOLOGY

By

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CERTIFICATE

This is to certify that the thesis entitled, "***The rheological study of slurry containing ball clay and using different deflocculant concentration & preparation of ceramic porous body using different volume concentration of ball clay & wheat flour***" submitted by **Nitesh Kumar Agrawal (111CR0002)** in partial fulfilment of the requirements for the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela, is a bonafide and authentic research work carried out by them under our supervision and guidance over the last one year (2014-15).

To the best of our knowledge, the work embodied in this thesis has not been submitted earlier, in part or full, to any other university or institution for the award of any Degree.

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“A teacher can teach a student a lesson for a day if he could teach practically; he would remember the learning process throughout his life”

The knowledge I gained and skill acquired during the whole course of the project working at the laboratory, Ceramic Engineering Department, will stay with me for life long.

I would like to convey our heartfelt gratitude and regards to my project supervisor **Prof. Swadesh Kumar Pratihar**, Department of Ceramic Engineering, National Institute of Technology, Rourkela for his outstanding guidance and for giving me such a mind stimulating and innovative project. He has always bestowed parental care upon us and evinced keen interest in solving my problems. An erudite teacher, a magnificent personality and a strict disciplinarian, I consider myself fortunate to have worked under his supervision.

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ABSTRACT

The rheological study of the slurry containing ball clay and using different deflocculant (Sodium Silicate) concentration has been done. Slurry with different solid loading are prepared by taking ball clay and water in different proportions with solid loading 30%, 35% and 40% (by volume). Deflocculant with different concentrations are mixed with different slurries (with different solid loading). Deflocuculant with 5 different proportions was taken. Defloculant with 0.25%, 0.5%, 0.75%, 1 % and 1.25% (weight %) of the clay were taken and poured in a plastic bottle with alumina balls for wet milling for 12hrs. Rheology of the slurry was then studied by measuring the viscosity, sedimentation height and zeta potential of the slurry.

Further with this optimized solid loading (30 Vol% Solid Loading) and the amount of deflocculant (0.85 wt. % of Ball clay), ceramic porous body was prepared with this fixed solid loading, but varying the ratio of ball clay and wheat flour. In this fixed solid loading, the wheat flour and ball clay was varied in the ratio 10:90, 20:80, 30:70, 40:60 and 50:50 by volume.The different slurry was prepared with varying ratio of wheat flour and ball clay as mentioned above. In this slurry, the amount of deflocculant was fixed 0.85% (wt %) of the total ball clay taken. These slurries were mixed through pot milling for 12 hours. Then the slurry was casted in a metal mold and kept at drier for 24 hours to get a cylindrical shaped body which was later fired at a different temperature. Different samples were fired at different temperatures to get the final ceramic porous body. Samples were fried at 1100^0C, 1200^0C and 1300^0C. Then characterization of the prepared ceramic porous body was done by determining Drying Shrinkage, Firing shrinkage, Apparent Porosity, Bulk Density and Cold Crushing strength.

Keywords: Ball clay slurry, Deflocculant concentration, Solid Loading, Double Layer thickness, Cation Exchange, Viscosity, Zeta Potential, Sedimentation Height, Ceramic Porous Body, Wheat flour, Swelling of wheat flour, Drying shrinkage, Firing shrinkage, Apparent Porosity, Bulk Density and Cold Cushing Strength.

CHAPTER 1

INTRODUCTION

Rheology is the science of deformation and flow. The knowledge of rheological behaviour is essential for the selection and designing of equipment for storing, pumping, transporting, milling, atomizing and forming a ceramic system. The rheological study is inevitable in the research and development of the slurry system. [1] The rheological properties of slip during slip casting is very important as it is a main parameter which controls the flow behaviour during casting, settling behaviour and properties of casting. Ceramic slurries and pastes are relatively complex and poorly characterized. The inter-particle spacing depends widely on the solid loading, the state of dispersion and the particle packing. The rheological property of the slip depends on the physical and chemical property of the raw material and the conditions under which the slip is prepared. The rheological properties of the slip affect properties of cast behaviour, casting time, etc. The important parameters which affect the rheological behaviour of the slurry are the chemical and mineralogical composition of raw materials, viscosity, particle shape and size, temperature, time, type of mixing, pH of the slurry. During slurry preparation, modification of particular parameter is done to achieve desired properties. Slip viscosity effectively controls the properties of ceramic casting slips. The quality and thickness of the cake formed can be changed by changing the slip viscosity. The water used in the slip casting process must be free from ions which affect the colloidal properties of the particles present in the suspension. As with the change in concentration of these ions, the rheological properties of the slurry changes depending whether it does a flocculating or deflocculating action. [2]

Ball clay and wheat flour are the two raw materials which are easily available and economical. Hence, the study of the rheology of china clay and ball clay is more reasonable and cost effective. Moreover, china clay and ball clay are used as a basic material for ceramic slurry preparation. Hence, their rheology should be studied as it helps in deciding many primary ceramic industrial applications.

Deflocculant is a chemical additive used to reduce the viscosity of the suspension. It prevents the flocculation of the suspended particles. Deflocculant are substances which prevent flocculation by increasing zeta potential and therefore, the repulsive forces between particles. The electrical potential between the surface and the bulk of the solution drops gradually away from the surface across a distance called double layer. The potential at the slippage plane, somewhere in between the double layer is called zeta potential. [1]

$Na_2O.nSiO_2$ (Sodium silicate)

It is one of the most effective and reliable deflocculant used in slip casting of different slurries. It is cheap and easily available which increases its demand. The ratio of SiO2 to Na2O in sodium silicate can vary from 3.75:1 to 1:1 and is available in the liquid or solid form. Sodium silicate increases the pH of the suspension, due to hydrolysis and the silicon separates out in the form of colloidal silica, which performs as a protective colloid, according to the following reaction:

$$Na_2O.nSiO_2 + H_2O \rightarrow nSiO_2 + 2Na^+ + 2OH^-$$

The research interest in the field of porous ceramics has increased in the recent decade due to their well controllable and customized microstructural features like porosity and pore size distribution. Low density in combination with the high strength, low thermal conductivity, high specific surface area, and high permeability, etc. is the unique characteristics of porous ceramics [1]. These properties make them more suitable for a wide range of technical applications which include catalyst supports, filters for molten metals and diesel engine exhaust, burners, biomedical device, kiln furniture and high-temperature thermal insulation, etc. [3].

Starch consolidation casting (SCC) belongs to the family of sacrificial template method for porous ceramics fabrication. The swelling/gelling property of wheat flour on hydration helps in body consolidation and dewatering processes. Porosity in the sample developed in the subsequent firing process due to burning out of the wheat flour. The porosity of the porous body depends on the wheat flour amount; viscosity of the slurry, particle size distribution of the ceramic powder, etc. This technique can fabricate a sample with a maximum of 60-70% porosity.

The strength of the porous ceramics depends on the porosity and more preciously the extent of bonding between the neighbouring ceramic particles (densification). Densification decreases the porosity of the sample. On the other hand, the porosity of the sample increases with the increase in pore former (sacrificial phase) content, in this case, wheat flour content in the precursor slurry.

The present thesis contains six chapters. Chapter 1 (this chapter) introduces the subject and content of the thesis. Detailed literature review is present in chapter 2. This chapter summarizes the importance of rheological study, porous ceramics, different fabrication techniques, and applications. Chapter 3 presents the procedures and the experimental work that has been carried out throughout this project. The flow chat of the experimental procedure has been presented in this chapter. The results and discussion of current research detailed in Chapter 4. This chapter contains all the results obtained from the rheological study of ceramic slurry along with the characterization of prepared ceramic porous body. All the graphs has been explained in this section. Chapter 7 summarizes the main findings and the conclusions of the present study.

CHAPTER 2

LITERATURE REVIEW

Adriano Michael Bernardin et al. [4] studied of the rheological behaviour of porcelain tile slurries produced by wet milling. They analysed the deflocculation of slurries using two different types of deflocculants (sodium meta silicate and sodium silicate). The slurry rheology was studied by observing the change in viscosity as a function of deflocculant. After the analysis, they concluded that sodium silicate is more effective than Sodium Meta Silicate. The best results for silicate were found in SiO_2/Na_2O ratio 2:1 and that of Metasilicate 3:1. This study shows the importance of viscosity control of samples in the ceramic industry. They also found to know that the rheological behaviours of the ceramic suspensions are not only affected by the deflocculant taken, but also by the hardness of water and pH and different characteristic of particles e.g.:- shape, particle size.

Himanshu Desai [5]studied the rheological behaviour of clay water system with different surfactants, viz.nonionic triton X-100 (TX -100), non-ionic sodium dodecyl benzene sulfonate (SDBS) and cationic acetyl pyridinium bromide (CPB) surfactant on pyrophyllite – water slurry using rotational cone and plate BOHLIN – VISCO 88 viscometer. It was observed that the addition of cationic surfactant to pyrophyllite slurry caused an increase in viscosity and then a decrease in viscosity due to charge reversal. However, in case of non-ionic surfactant the viscosity first increased after which it remained constant.

F.N. Shi et al. [6] found out a new procedure for obtaining full shear stress- shear rate flow rate curve for different unstable slurries using the single bobbin- Debex on-line viscometer. They showed that torque coefficient data from a variety of Newtonian fluids and non-Newtonian slurries fell on a single curve, which was characteristic for a particular instrument design.

C. M.Gomes et al. [7] Studied the effect of two different types of sodium silicate on the rheological behavior of triaxial ceramic suspensions with 40 wt. % solid loading for both the composition and determined the minimum amount of deflocculant required for stabilizing the slurry. It was noted that an increase in alkalinity of the sodium silicate resulted in lowering of the viscosity of the dispersion.

R.R. Klimpel [8] found out that wet grinding of materials in most industrial grinding devices can be significantly changed by slurry rheology. He concluded that pulp density, the level of fineness present and pulp chemistry are important to maximize the throughput.

F.H. Norton [9] carried out research on especially samples prepared from natural clay. The study was made on kaolinite samples with known particle size, known organic content and known controlled adsorbed ions. The effect of any one variable on the plasticity and that workability of clay slip was studied. On basic information, water clay system is needed to understand the complex behaviour of natural clays.

Pores present in the porous structure are generally classified into three groups depending on their sizes namely microporous, mesoporous and macroporous whose sizes are in the range of less than 2 nm, between 2-50 nm and above 50 nm respectively [10]. Micro and mesoporous ceramic materials are used as molecular sieves [11], in catalysis [12] and controlled release applications [13, 14] whereas the use of macroporous ceramics applications starts from traditional ceramics like roof tiles to advanced technical ceramics in medicine and automobile engines [15].

The development of the porous filters satisfied the requirements like recovery of the methane from mines, removal of carbon dioxide and hydrogen sulfide from nature gas, recovery of hydrogen in petroleum refinery operation. Foundry industry uses porous filters for hot metal filtration [16]. The catalyst supported porous materials have been employed in the mass transfer of the catalytic combustion [17], combustion in-situ in underground reservoirs for enhanced oil recovery, heat transfer devices, diesel particulate filters [18] and reduction of hazardous combustion products. Porous ceramics also used in sensors, battery materials, thermal protection materials, and biomedical applications [19].

Development of porous ceramics with high porosity with adequate strength has received much attention in the recent decades due to their tailored microstructure and unique properties. Pore morphology (i.e, porosity, pore size distribution) together with the unique properties like low density, high strength, low thermal conductivity, high thermal stability, high specific surface area, and high permeability are unique characteristics of porous ceramics. These properties are suitable for a wide range of technical applications which include catalyst supports, filters for molten metals, hot gases and ion exchange, burners, biomedical device, kiln furniture and high temperature thermal insulation etc., where the high temperature, corrosion resistant and wear resistant environments are involved.

CHAPTER 3

EXPERIMENTAL WORK

3.1 RHELOGICAL STUDY OF BALL CLAY WITH DEFLOCCULANT

3.1.1 Clay Slurry Preparation

Clay slurries were prepared by using ball clay and sodium silicate as a deflocculant in different weight ratio. Different weight of ball clay was taken to get solid loading of 30 Vol%, 35 Vol% and 40 Vol%. For every solid loading, five different slurries were prepared by adding deflocculant in different weight ratio. The amount of deflocculant added was 0.25 wt. %, 0.5 wt. %, 0.75 wt. %, 1.0 wt. % and 1.25 wt. % of the amount of ball clay taken. Every composition of ball clay and deflocculant was wet milled in a pot mill for 12 hours using alumina grinding media. For every composition, the volume of slurry was kept 100ml.

<table>
<tr><th colspan="6">BATCH COMPOSITION</th></tr>
<tr><th>SOLID LOADING (in vol %)</th><th>VOLUME OF WATER (in ml)</th><th>VOLUME OF BALL CLAY (in ml)</th><th>AMOUNT OF BALL CLAY (in gm)</th><th colspan="2">AMOUNT OF DEFLOCCULANT (in gm)</th></tr>
<tr><td rowspan="5">30 Vol %</td><td rowspan="5">70 ml</td><td rowspan="5">30 ml</td><td rowspan="5">77.04 gm</td><td>0.25 Wt.%</td><td>0.1926</td></tr>
<tr><td>0.50 Wt.%</td><td>0.3852</td></tr>
<tr><td>0.75 Wt.%</td><td>0.5778</td></tr>
<tr><td>1.00 Wt.%</td><td>0.7704</td></tr>
<tr><td>1.25 Wt.%</td><td>0.9632</td></tr>
<tr><td rowspan="5">35 Vol %</td><td rowspan="5">65 ml</td><td rowspan="5">35 ml</td><td rowspan="5">89.88 gm</td><td>0.25 Wt.%</td><td>0.2247</td></tr>
<tr><td>0.50 Wt.%</td><td>0.4494</td></tr>
<tr><td>0.75 Wt.%</td><td>0.6741</td></tr>
<tr><td>1.00 Wt.%</td><td>0.8988</td></tr>
<tr><td>1.25 Wt.%</td><td>1.1235</td></tr>
<tr><td rowspan="5">40 Vol %</td><td rowspan="5">60 ml</td><td rowspan="5">40 ml</td><td rowspan="5">102.72 gm</td><td>0.25 Wt.%</td><td>0.2568</td></tr>
<tr><td>0.50 Wt.%</td><td>0.5136</td></tr>
<tr><td>0.75 Wt.%</td><td>0.7704</td></tr>
<tr><td>1.00 Wt.%</td><td>1.0272</td></tr>
<tr><td>1.25 Wt.%</td><td>1.2841</td></tr>
</table>

Table.1- Batch composition for clay slurry with Sodium Silicate as deflocculant

3.1.2 Slurry Characterization

All the prepared slurries after wet milling (for 12 hrs in pot mill) were subjected to characterization. And the slurry characterization is done by doing following tests-

1. Viscosity Measurement with different shear rates and Shear – Stress Rate Behaviour both during the upswing and downswing.
2. Zeta Potential Measurement
3. Settling height with time

3.1.2.1 Viscosity Measurement with different shear rates and Shear – Stress Rate Behaviour both during the upswing and downswing.

The slurries were subjected to shear stress in a concentric Cylinder Rheometer Model Rheolab, Anton Parr at shear rates 1-100 (sec-1) and the shear stress and viscosity were noted.

The measurement was done both during upswings (i.e. 1- 100sec-1) and down swing (100-1sec-1) mode.

From the obtained graphs, the change in viscosity with change in shear rate can be observed for all the compositions.

3.1.2.2 Measurement of Zeta Potential

The zeta potential of the slurry was measured using the Malvern Zetasizer. The value of zeta potential value was plotted against the deflocculant concentration for all the solid loading.


Figure-1 (A) Concentric Cylinder Rheometer Model Rheolab, Anton Parr; (B) ZetaSizer (Malvern)

3.1.2.3 Measurement of Settling Height

The settling height of the slurries was measured after doing the rheology test. The slurries were poured into a vertical measuring cylinder of volume 100 ml. The slurry was initially filled up to the 50ml mark of that measuring cylinder and then the height of the slurry was noted at regular intervals to get the settling height. Similarly, it is done for all the slurries. Finally, the difference in height of the slurry was plotted against time.

3.2 PREPARATION OF POROUS CERAMICS WITH BALL CLAY & WHEAT FLOUR

3.2.1 Clay Slurry Preparation with wheat flour

Clay slurries with wheat flour were prepared by using ball clay, wheat flour in different weight ratio and sodium silicate as a deflocculant with a constant amount (0.85 Wt. % of total ball clay taken). The different volume ratio of ball clay and wheat flour was taken to get a constant solid loading of 30 Vol%. Five different slurries were prepared by adding a different volume ratio of ball clay and wheat flour. Wheat flour and ball clay are taken in five different volume ratios- 10:90, 20:80, 30:70, 40:60 and 50:50.The amount of deflocculant added was 0.85 wt. % of the amount of ball clay was taken which was kept constant. Every composition of ball clay and wheat flour, the slurry was wet milled in a pot mill for 12 hours using alumina grinding media. For every composition, the volume of slurry was kept 100ml.

BATCH COMPOSITION (SOLID LOADING 30 VOL %)						
RATIO OF WHEAT FLOUR TO BALL CLAY (in vol %)	VOL OF WATER (in ml)	VOL OF WHEAT FLOUR (in ml)	VOL OF BALL CLAY (in ml)	AMOUNT OF BALL CLAY (in gm)	AMOUNT OF WHEAT FLOUR (in gm)	AMOUNT OF DEFLOCCULANT (in gm)
10:90	70 ml	3 ml	27 ml	69.336 gm	4.674 gm	0.589 gm
20:80	70 ml	6 ml	24 ml	61.632 gm	9.348 gm	0.523 gm
30:70	70 ml	9 ml	21 ml	53.928 gm	14.022 gm	0.458 gm
40:60	70 ml	12 ml	18 ml	46.224 gm	18.696 gm	0.392 gm
50:50	70 ml	15 ml	15 ml	38.520 gm	23.371 gm	0.327 gm

Table.2- Batch composition for clay slurry with wheat flour & Sodium Silicate as deflocculant (0.85 wt. % of Ball Clay)

3.2.2 Metal Casting of clay slurry containing wheat flour

The Casting of these slurries was done in cylindrical metal molds. Slurries after wet milling (for 12 hrs in pot mill) were taken out and poured in cylindrical metal molds up to a certain level. The height of the cylindrical metal molds was taken 4cm and diameter were 2cm. All the metal molds were properly cleaned and lubricant was applied inside the mold which is necessary to prevent any defects during demolding of the ceramic porous body. Now the slurries were poured in all the metal molds. The slurry of each composition was poured in six metal molds to get six samples which will be used for characterization. Similarly, the rest of the slurries were also poured in all of the remaining metal molds.

3.2.3 Swelling of wheat flour

Wheat flour was a property of swelling when water is added in it. Swelling of wheat flour is important so as to obtain the porosity inside the ceramic body. Wheat flour absorbs some amount of water and it expands which leads to increase in volume. For this, the swelling test of the wheat flour is done to measure the change in volume. Some finite amount of wheat flour was taken inside a cylindrical measuring cylinder (100ml), filled up to a certain height (10ml) and the cylinder was filled with water up to 100ml. Now the cylinder was kept inside the drier at 80^0C and at regular intervals (after every 15 mins) the rise in the height of the wheat flour was noted till the saturation limit is obtained where the height is no more increasing with time. Similarly, the same experiment was repeated at 90^0C to get the change in volume of wheat flour with time at two different temperatures. After noting down the heights of both the experiments, the graph between Δ V/ V with time was plotted for both 80^0C and 90^0C. The physical appearance of the sample before and after swelling has shown in the Figure 2.

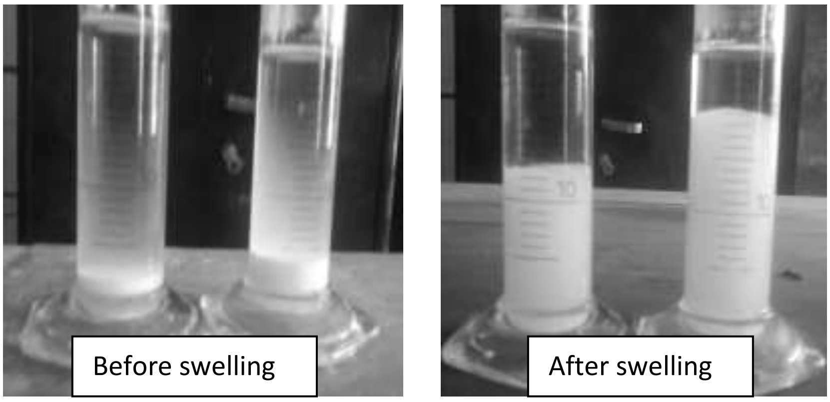


Figure 2- Physical appearance of the wheat flour sample before and after swelling

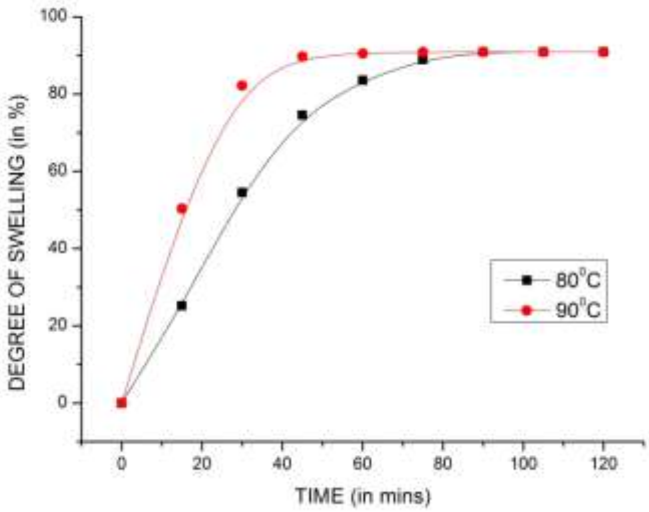


Figure 3 Swelling behaviour of wheat flour as a function of time with effect of temperature

The swelling behavior of wheat flour as a function of time, temperature has been provided in Figure 3. Figure 3 shows the swelling behavior of wheat flour as a function of time and temperature. The wheat flour undergoes a rapid swelling during the initial period thereafter that it slows down. The volume expansion of the wheat flour on heating was due to the uptake of water by molecules of wheat flour. The swelling behaviour, especially the amount of maximum swelling on hydration remains constant as a function of temperature. The only difference observed lies with the time required to complete swelling. It increases with the decrease in swelling (reaction) temperature. Swelling rate increases with increases with a rise in temperature. Wheat flour showed a maximum swelling of 90.91%. It is worthy to note that the total volume of the wheat flour-water system remains constant during the swelling reaction. Thus, the study concludes that the swelling of wheat flour is equal to the water taken by starch during the swelling process.

3.2.4 Drying and Demolding

After casting the slurries into the metal molds, the molds were kept inside the drier (2 hrs) at 80^0C for swelling of the wheat flour to occur. Within this time, the swelling of wheat flour occurs which leads to absorption of water by the wheat flour. After keeping the molds inside the drier for 2 hrs at 80^0C, the temperature of the drier was gradually increased up to 100^0C for complete drying. The molds were dried at 100^0C for 24 hours for complete removal of physical water from the samples. After 24 hours of drying, the slurry has already taken the shape of the cylindrical metal mold and now can be easily removed from the metal molds. All the samples were taken out of the metal mold and the diameter of every sample was noted to calculate the drying shrinkage.

3.2.5 Characterization of the prepared ceramic porous body

After taking out all the samples from the metal molds, the characterization of all the samples has been done. And the characterization is done by doing following tests-

1. Drying shrinkage
2. Firing shrinkage
3. Porosity Measurement
4. Cold Crushing strength

3.2.5.1 Drying shrinkage

The molds were dried at 100^0C for 24 hours for complete removal of physical water from the samples. After 24 hours of drying, the slurry has already taken the shape of the cylindrical metal mold and now can be easily removed from the metal molds. All the samples were taken out of the metal mold and the diameter of every sample was noted to calculate the drying shrinkage. The initial diameter of the metal mold was 2cm and the final diameter of all the samples was measured by digital Vernier calliper. Drying shrinkage was calculated by taking the difference in initial diameter and final diameter.

3.2.5.2 Firing shrinkage

After calculating the drying shrinkage, the samples were then fired at different temperatures. Out of 6 samples of each composition, two samples from each composition were kept in the furnace for firing at 1100^0C, 1200^0C and 1300^0C with a soaking time of 2 hrs at final temperature and at 650^0C for 1 hr. The firing rate was kept constant of 3^0C/min. During firing, all the wheat flour was burned out along with chemically reacted water present inside the samples. After final completion of firing, the diameter of all the samples was noted down to calculate the firing shrinkage.

3.2.5.3 Porosity Measurement

The porosity of the prepared ceramic porous samples was measured by Archimedes principle. Dry weights of all the samples were taken, and then the samples were kept inside a beaker filled with Kerosene. Then the system placed in a vacuum desiccator for about three hours under vacuum. The suspended and soaked weight of the samples was measured using a weight balance. After measuring all the weights, porosity and density of the samples were calculated by using the formulae given below:

$$Apparent\ Porosity = \frac{Suspended\ Weight - Dry\ Weight}{Suspended\ Weight - Soaked\ Weight} \quad (3.1)$$

$$Bulk\ Density = \frac{Dry\ Weigth\ \ X\ Density\ of\ Water}{Suspended\ Weight - Soaked\ Weight} \quad (3.2)$$

$$Relative\ Density = \frac{Bulk\ Density}{Theoretical\ Density} X\ 100 \quad (3.3)$$

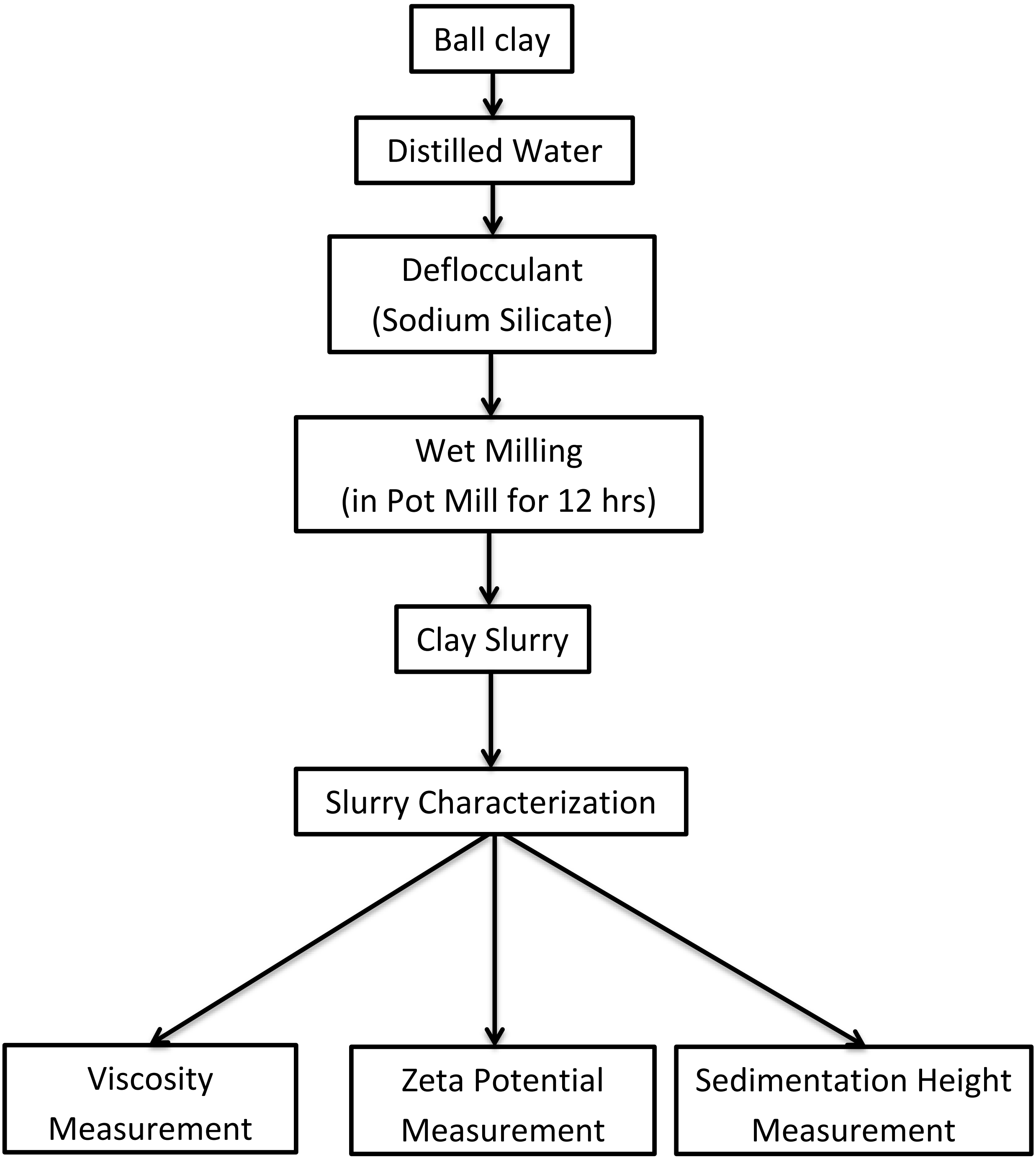
$$Total\ Porosity = 1 - Relative\ Density \quad (3.4)$$

3.2.5.4 Cold Crushing Strength

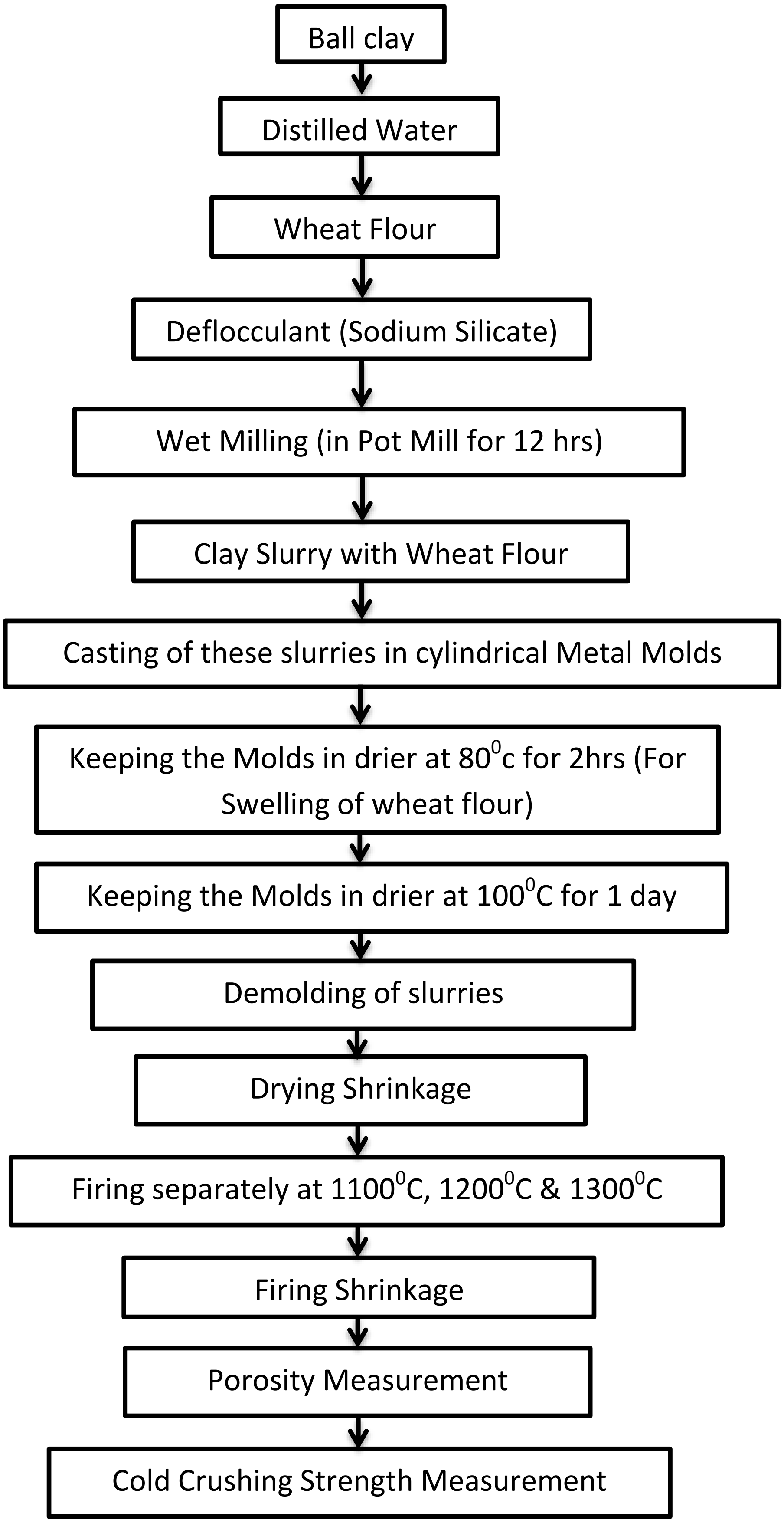
The compressive strength of the fired samples was measured by using a universal testing machine (Enhanced Digital Indicator, MUCTM, Aimil) crushed under parallel plates with a crosshead speed of 0.5mm/min.

Figure 4- (A) Samples for Apparent porosity Measurement; (B) Universal testing machine (Enhanced Digital Indicator, MUCTM, Aimil)

FLOW CHART OF RHEOLOGICAL STUDY OF BALL CLAY WITH DEFLOCCULANT



PREPARATION OF POROUS CERAMICS WITH BALL CLAY & WHEAT FLOUR



CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effect of solid loading on Zeta Potential for varying amount of Sodium Silicate as deflocculant

The zeta potential of the slurry was measured using the instrument Malvern ZetaSeizer. The value of zeta potential value has been tabulated below for all the slurries. And a graph of zeta potential value was plotted against the deflocculant concentration for all the solid loading.

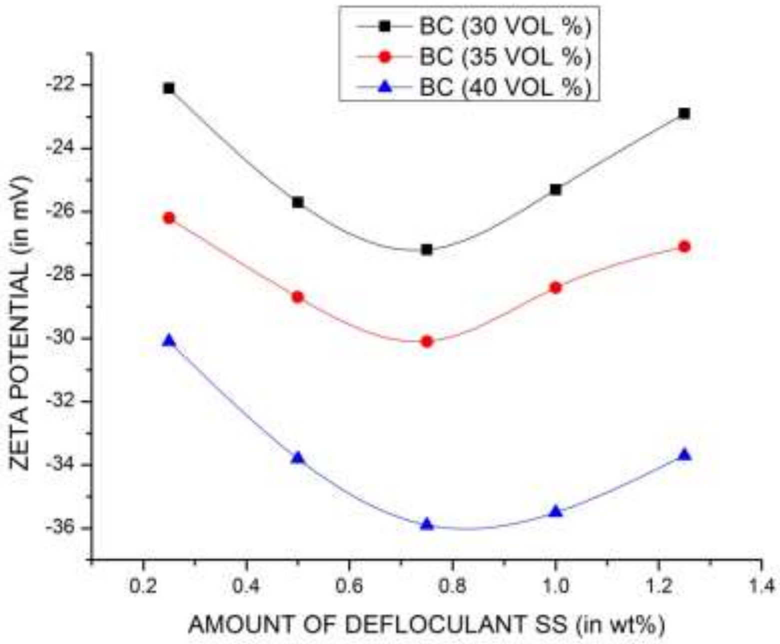


Figure 5- Effect of solid loading on Zeta Potential for varying amount of Sodium Silicate

The effect of solid loading on Zeta Potential for varying amount of Sodium Silicate has been plotted in the figure 5. From the above graph, it can be seen that Zeta Potential first increases on increasing the amount of deflocculant concentration and then decreases with increasing the amount of deflocculant concentration. Initially on increasing the amount of deflocculant, double layer thickness increases, which increase the zeta potential, but on after adding more and more amount of deflocculant, charge accumulation takes places on the surface of clay particle due to which particle-particle repulsion takes place. Therefore, the zeta potential decreases after a certain amount of deflocculant addition. But as solid loading increases, the more solid loading will have more negative charges on the surface of clay particle and double layer thickness increases.

4.2 Effect of time on Sedimentation Height for constant amount of Sodium Silicate as deflocculant

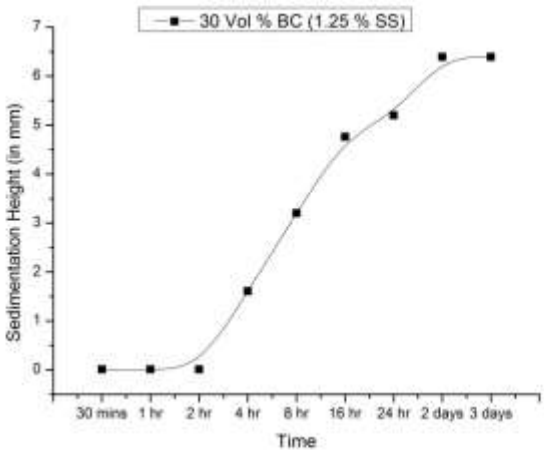


Figure 6- Effect of time on Sedimentation Height for constant amount of Sodium Silicate

Figure 6 shows the effect of time on Sedimentation Height for constant amount of Sodium Silicate. From the above figure, it can be easily seen that as time increases the sedimentation height of the slurry kept in measuring cylinder increases. After keeping the slurry for long time, the sedimentation height attains a maximum value and it remains constant. It can be explained as time increases more amount of clay particles in the slurry are getting settled due to the gravity.

4.3 Effect of solid loading on Sedimentation Height for varying amount of Sodium Silicate as deflocculant

Final Sedimentation Height (After 3 days) -

AMOUNT OF DEFLOCCULANT	SEDIMENTATION HEIGHT (in mm) (30 Vol %)	SEDIMENTATION HEIGHT (in mm) (35 Vol %)	SEDIMENTATION HEIGHT (in mm) (40 Vol %)
0.25%	0	0	0
0.50%	5.611	4.799	0
0.75%	4.799	4.011	0
1.00%	4.799	4.003	3.203
1.25%	6.395	4.799	4.003

Table.3- Final Sedimentation Height on varying amount of Sodium Silicate (SS) for all solid loading

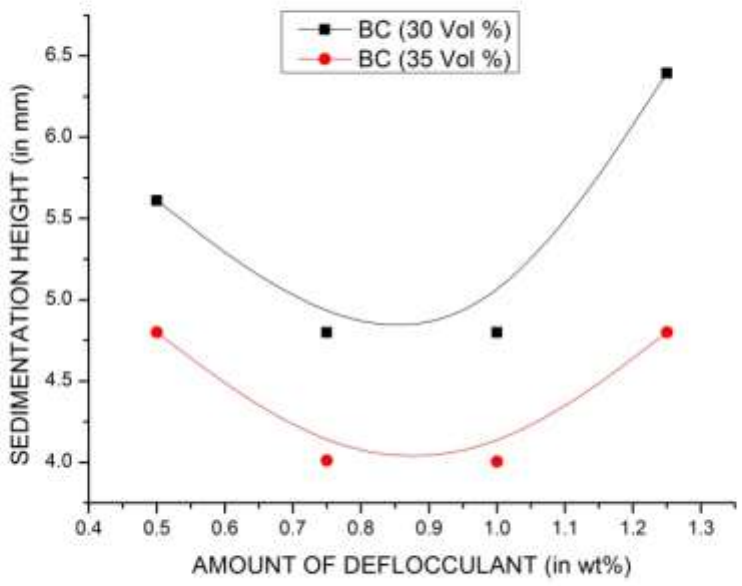


Figure 7- Effect of solid loading on Sedimentation Height for varying amount of Sodium Silicate

The effect of solid loading on Sedimentation Height for varying amount of Sodium Silicate has been showed in the figure 7. The sedimentation height of the slurry first increases and then decreases with the increase in the amount of Deflocculant. Initially when the amount of Deflocculant was increased, the Zeta Potential value increases which stabilises the slurry due to which the sedimentation height decreases. But after a certain value of Deflocculant, the Zeta Potential decreases which results in instability of the slurry and leads to increase in the sedimentation of the ceramic slurry.

4.4 Effect of Solid Loading on viscosity of the slurry for varying amount of Sodium Silicate as deflocculant

From the above graph, it can be seen that viscosity decreases with increasing the amount of deflocculant. By increasing the solid content in the slurry, the viscosity of the slurry increases. At certain values of deflocculant concentration, the viscosity has the minimum value. Viscosity first decreases with increase in deflocculant concentration, reaches a minimum level.

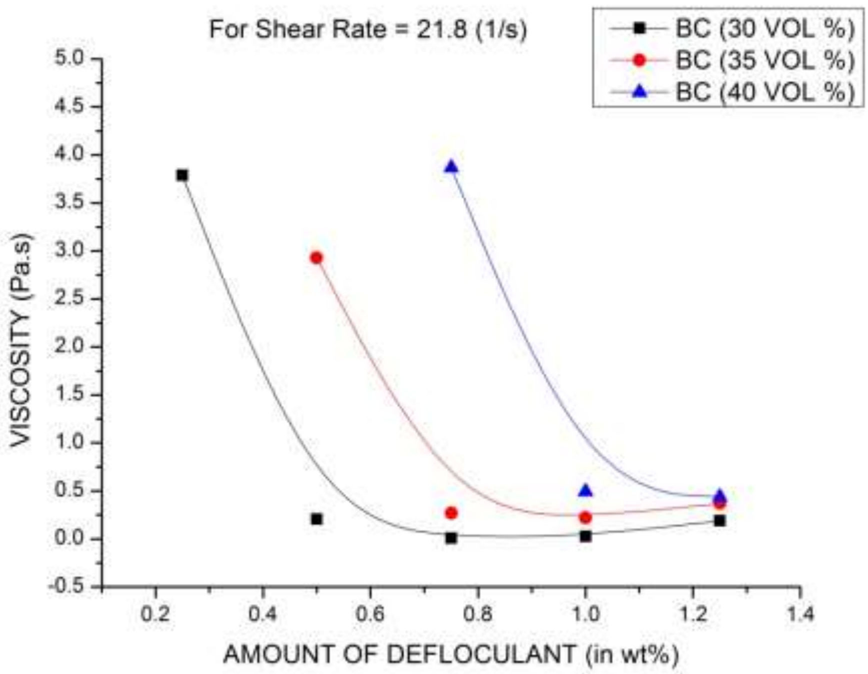


Figure 8- Effect of Solid Loading on viscosity of the slurry

Figure 8 shows the effect of Solid Loading on viscosity of the slurry. From the above graph, it can be seen that the viscosity of the ceramic slurry first decreases and then increases with increase in the amount of Deflocculant. The graph can be explained as the zeta potential value of the slurry first decreases and then increases with increase in the amount of Deflocculant. As the value of zeta potential increases, the double layer thickness increases which helps in increasing the stability of the clay slurry. Due to this stabilization the viscosity of the slurry decreases and attains a minimum for a certain amount of Deflocculant which cab seen from the above graph. But when the amount of Deflocculant was further increased, the Zeta potential value increases, accumulation of charges makes the slurry unstable due to which the viscosity of the slurry starts increasing.

4.5 Effect of Shear Rate on viscosity of the slurry for constant amount of Sodium Silicate as deflocculant (For 30 Vol% Solid Loading)

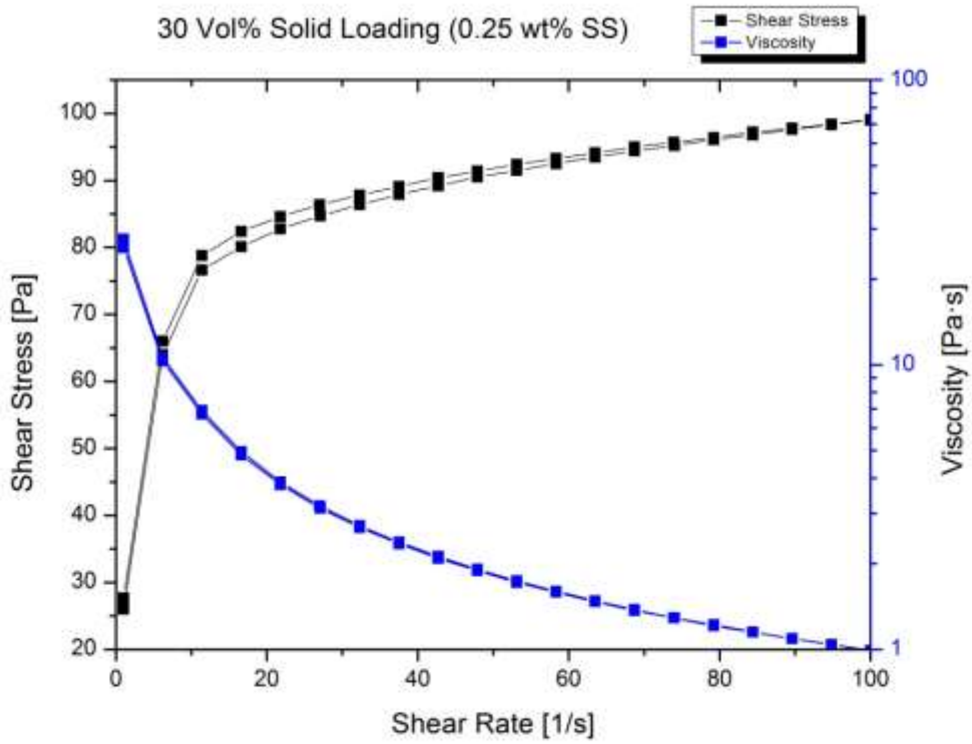


Figure 9- Effect of Shear Rate on viscosity of the slurry for constant amount of Sodium Silicate

The effect of Shear Rate on viscosity of the slurry for constant amount of Sodium Silicate (30 Vol. % Solid Loading & 0.25 wt. % Sodium Silicate) has been shown in the figure 9. From the above graph, it can be seen that viscosity decreases with increasing the shear rate. In doing the experiment, as the shear rate increases, the torque applied to the slurry increases, which decrease the viscosity of the slurry. And the Shear stress increases with increase in the shear rate and hysteresis loop formed by shear rate shows the shear thinning mechanism.

4.6 Effect of Shear Rate on viscosity of the slurry for varying amount of Sodium Silicate as deflocculant

From the above graph, it can be seen that at a higher deflocculant concentration on changing the shear rate there is no change in viscosity. As at higher deflocculant concentration, the slurry has sufficient flowability which has no effect on changing deflocculant on higher deflocculant concentration. It can be seen from the graph that the slurry has a minimum viscosity after 0.8wt. % deflocculant concentration.

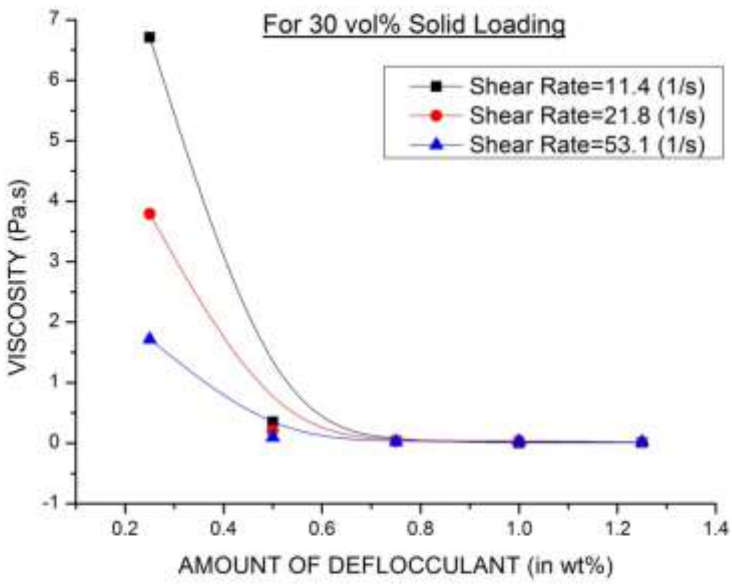


Figure 10- Effect of Shear Rate on viscosity of the slurry

4.7 Effect of amount of wheat flour on Drying Shrinkage for constant amount of Sodium Silicate as deflocculant

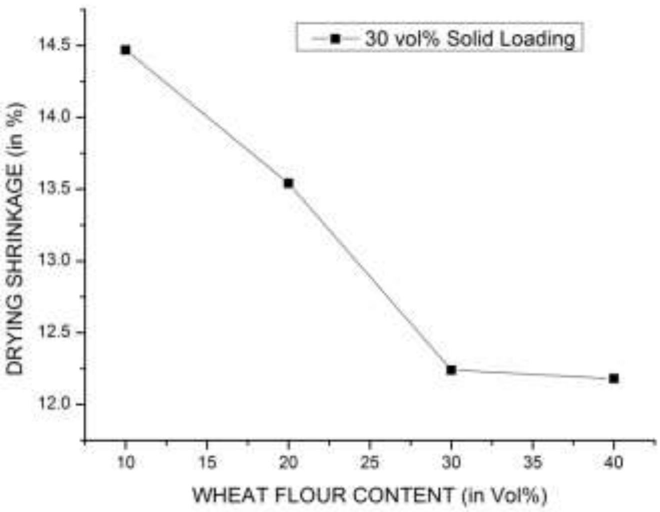


Figure 11- Effect of amount of wheat flour on Drying Shrinkage for constant amount of Sodium Silicate

In the above graph, it can be seen that the drying shrinkage of the samples decreases with increasing the amount of wheat flour in the slurry. As the amount wheat flour increases, it absorbs more water and swells. Thus, after drying at 100^0C, free water is evaporated out while the absorbed water is left in the sample which decreases the drying shrinkage.

4.8 Effect of amount of wheat flour on Firing Shrinkage for constant amount of Sodium Silicate as deflocculant

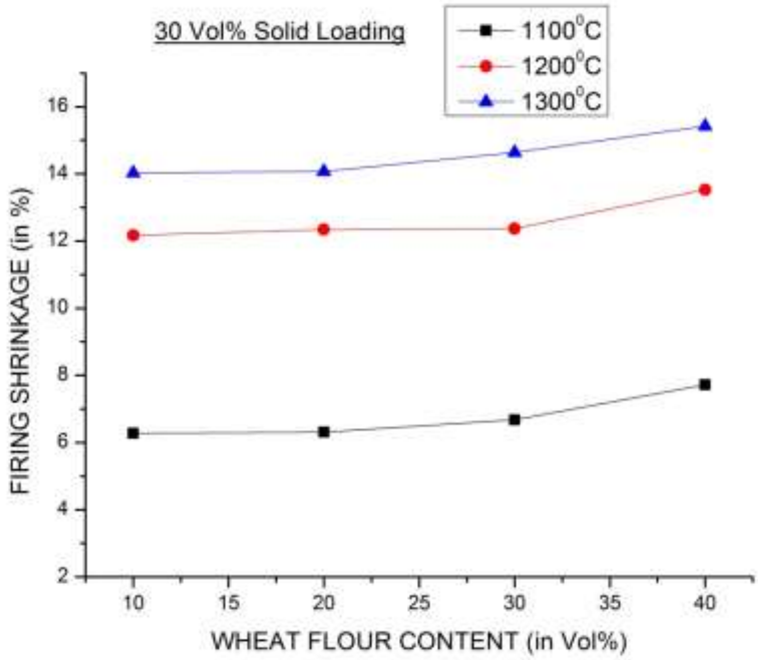


Figure 12- Effect of amount of wheat flour on Firing Shrinkage for constant amount of Sodium Silicate

In the above graph, it can be seen that the frying shrinkage of the samples increases with increasing the amount of wheat flour in the slurry. After firing the samples (above 1000^0C), all the chemically reacted water and wheat flour burns out which gives the porosity inside the samples. Due to this complete burning of wheat flour, it gives more firing shrinkage.

4.9 Effect of amount of wheat flour on Apparent Porosity for constant amount of Sodium Silicate as deflocculant

In the below graph, it can be seen that the apparent porosity of the samples increases for increasing the amount of wheat flour in the slurry. After complete burning of wheat flour, the porosity increases and the sample have the maximum value of apparent porosity which is having more amount of wheat content in it.

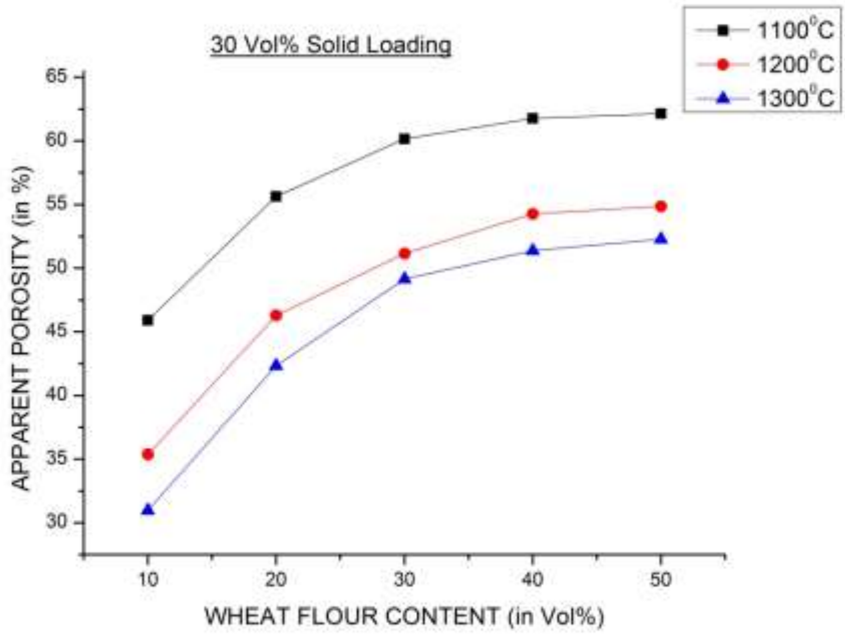


Figure 13- Effect of amount of wheat flour on Apparent Porosity for constant amount of Sodium Silicate

4.10 Effect of amount of wheat flour on Bulk Density for constant amount of Sodium Silicate as deflocculant

In the above graph, it can be seen that the bulk density of the samples decreases with increasing the amount of wheat flour in the slurry. Similarly for the bulk density the reason will be same as above.

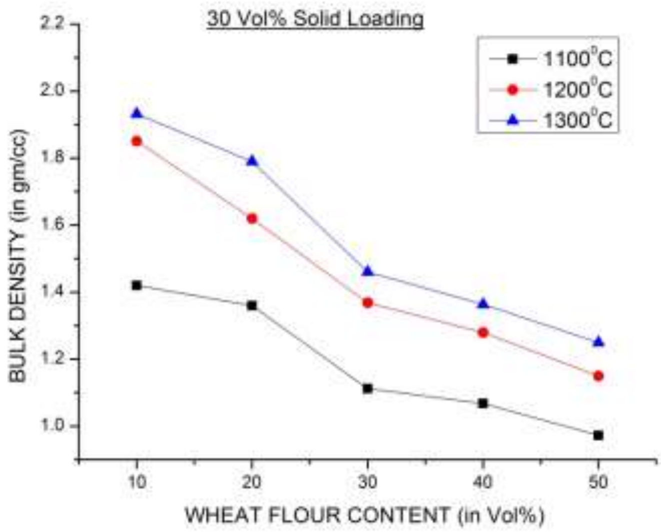


Figure 14- Effect of amount of wheat flour on Bulk Density for constant amount of Sodium Silicate

4.11 Effect of amount of wheat flour on Cold Crushing Strength for constant amount of Sodium Silicate as deflocculant

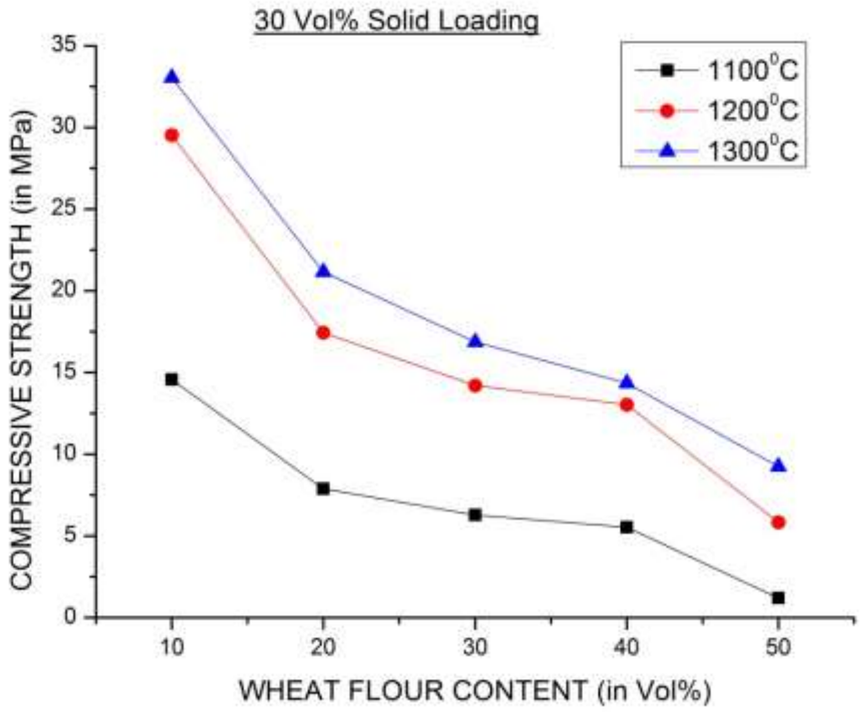


Figure 15- Effect of amount of wheat flour on Cold Crushing Strength for constant amount of Sodium Silicate

In the above graph, it can be seen that the cold crushing strength of the samples decreases with increasing the amount of wheat flour in the slurry. After complete burning of wheat flour, the porosity increases and the sample have the maximum value of apparent porosity which is having more amount of wheat content in it. Thus, on increasing the amount of wheat flour, porosity increases due to which the value of cold crushing strength decreases.

CHAPTER 5

CONCLUSIONS

CONCLUSIONS

The present research work discussed the rheological study of the slurry containing ball clay and different amount of deflocculant and preparation of the porous ceramics by using ball clay and wheat flour through metal casting. Characterization of the ball clay slurry has been done by doing different tests- Viscosity Measurement, Zeta Potential Measurement and Sedimentation Height. Studies show that the viscosity of the ball clay slurry decreases with increasing the deflocculant concentration, the Zeta potential value of the slurry first increases, then decreases with increasing the deflocculant concentration and sedimentation height first decreases, then increases with increase the deflocculant concentration.

Further, in this project, porous ceramics have been prepared by using the ball clay and wheat flour through metal casting. Characterization of the ball clay slurry containing wheat flour has been done by measuring- Drying shrinkage, Firing shrinkage, Porosity measurement and cold crushing strength. From the experiments, it was observed that drying shrinkage decreases on increasing the wheat flour content, firing shrinkage increases on increasing the wheat flour content, firing shrinkage increases on increasing the wheat flour content, apparent porosity increases on increasing the wheat flour content, bulk density decreases with increasing the wheat flour content and cold crushing strength decreases on increasing the wheat flour content. Thus, samples with porosity in the range 10-65% could be prepared by the above technique.

REFERENCES

1. J.S. Reed, “Deflocculants and Coagulants”, “Rheology Of Saturated Systems”

2. A. Evcin, S. Orencik, T. Kavas “Effects Of Inorganic Salts On Rheological Properties Of Slips”, J Key Engineering Vols. 264- 268(2004)Pp. 1609-1612

3. Digital Reference Database

4. A. M. Bernardin, M. C. Casagrande, H. G. Riella, “Rheological Behavior Of Porcelain Tile Slurries”, J Qualicer 2006

5. H. Desai, “Effect of Surfactants On Clay – Water Slurry Rheology”

6. F. N. Shi, T.J. Napier- Munn “Measuring The Rheological Of Slurries Using An On –Line Viscometer”, Int. J. Miner Process. 47(1996) 153-176

7. C.M. Gomes, J.P. Reis, S.L. Correia , A.P.N. Oliveira, D. Hotza, “ Influence Of Different Types Of Sodium Silicate In Compositions Of Triaxial Ceramics Using A Mixture Design Approach”, J Qualicer 2004

8. R.R. Klimpel, “The Selection of Wet Grinding Chemical Additives Based On Slurry Rheology Control”, Powder Technology 105(1999) 430-435

9. F.H. Norton, “Flow Properties Of The Kaolinite- Water System”

10. M.N.Rahaman, “Ceramic processing and sintering” pp. Marcel Dekker Inc, New York, 1998.

11. FatemeRezaei, Alessandra Mosca, Paul Webley, Jonas Hedlund and Penny Xiao, “Comparision of traditional and structural absorbents for CO2 separation by vacuum swing adsorption” Ind.Eng.Chem.Res. 49 (2010) 4832-4841.

12. AvelinoCorma, “From microporous to mesoporous molecular sieve materials and their use in catalysis” Chem. Rev. 97 (1997) 2373-2419.

13.Gustav Nordlund, Jovice Boon Sing Ng, Lennart Bergstrom and Peter Brzezinski, “ Amembrane reconstituted multisubunit functional proton pump on mesoporous silica particles” Am. Chem. Soc., vol.3 (2009) 2639-2646.

14.Jenny Andersson, Jessica Rosenholm, Sami Areve, and Mika Linden, “Influences of material characteristics on ibuprofen drug loading and release profiles from ordered micro and mesoporous silica matrices” Chem. Mater. 16 (2004) 4160-4167.

15. M.V.Twigg and J.T.Richardson “Theory and applications of ceramic foam catalysts” Trans IChemE, Vol 80 (2002).

16. J.Luyten, S.Mullens, I.Thijs, “Designing with pores synthesis and applications” KONA Powder and Partcle journal, 28 (2010).

17. Michele Ambrogio, Guido Saracco, Vito Specchia, “Combining filtration and catalytic combustion in particulate traps for diesel exhaust treatment,” Chem. Engg. Sci. 56 (2001) 1613-1621.

18.Z. Taslicukur, C. Balaban, N. Kuskonmaz, “Production of foam filters for molten metal filtration using expanded polystyrene,” J. Eur. Ceram. Soc. 27 (2007) 637-640.

19. J.M.Taboas, R.D.Maddox, P.H.Krebsbach, S.J.Hollister, “Indirect solid free form fabrication of local and global porous, biomimetic and composite 3D polymer ceramic scaffolds,” Biomaterials 24 (2003) 181-194